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## (54) Method for preparing implant surfaces

Verfahren zur Herstellung von Implantatoberflächen

Méthode de préparation des surfaces de corps d'implantation

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(56) References cited:

EP-A- 0 248 117

EP-A- 0 523 372

WO-A-88/06459

WO-A-93/12821

FR-A- 2 318 617

US-A- 5 071 351

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**Description**

[0001] The present invention relates to a method for preparing implant surfaces of metallic material, preferably titanium, using gas-discharge plasma with the aim of obtaining a well-defined and reproducible implant surface.

[0002] It is previously known permanently to anchor oral and extraoral prostheses in bone tissue. In order to avoid the prostheses loosening, a healing-in period is required with direct contact, i.e. exact adaptation, between the prosthesis and the bone tissue. Such an exact, enduring, adaptation permitting load-transfer is called osseointegration. That such an enduring adaptation and function can be achieved in clinical practice using load-bearing synthetic fixing elements was first demonstrated by Dr Brånemark and co-workers. They showed that screw-shaped fixing elements, so-called fixtures, of pure titanium were osseointegrated if a number of preconditions are fulfilled. The osseointegration principle developed by Professor Brånemark and co-workers has a very high success rate (more than 90 %) and has been used clinically since 1965. In all, several 100,000 fixtures have by now been implanted throughout the world.

[0003] The osseointegration principle is based not only on the implant being executed in pure titanium, but also on a first operation in which the fixture is inserted by an atraumatic technique, a healing phase of critical length without loading, and a second operation involving attachment of a loading element (the prosthesis part), i.e. a two-session process with an intermediate healing period without loading.

[0004] In order that the fixture heals well in the body tissue, it is necessary for a minimum of negative biological reactions to be induced, or, even better, for reactions which are predominantly positive to be induced. The structure and the chemical composition of the surface layer of the fixture have been found to be of crucial importance for these reactions.

[0005] Swedish Patent 79.02035-0 describes how improved results can be achieved by imparting a specific surface structure to the titanium-containing material in the fixture. The surface layer of the fixture consists of micro-cratered titanium dioxide, with the craters in the surface having a diameter of 10-1000 nm, preferably 10-300 nm.

[0006] The surface properties (structure and chemical composition) of the implant are thus important for its function since they determine how the tissue at the site of implantation will react. During manufacture of implants, therefore, continuous analyses are carried out to check that the surfaces of the components fulfil a given specification, inter alia with regard to microstructure, composition, purity and sterility. It is thus desirable that the production methods give reproducible results.

[0007] Nowadays, the titanium components are made in the following manner. Firstly, there is a monitored ma-

ching using computer-controlled workshop machines. This machining gives a surface having microscopic irregularities.

[0008] During the machining with cutting tools, oxidation of the workpiece takes place. The oxidation involves formation of titanium oxides. The machining also promotes the formation of a biologically correct surface structure of micro-cratered character (moon-surface type), in accordance with the abovementioned patent.

[0009] After the machining, the components are burried and inspected. After that, the surface is cleaned by means of a number of washing steps in different liquids. The washed components are then packed manually in glass ampoules which are sealed hermetically (by fusing with the aid of a flame). The content of the glass ampoule is then heat-sterilized, after which the sterile package is packed in a so-called blister pack made of plastic. The blister packs are then also sterilized, after which they are packed in cardboard boxes.

[0010] During the machining, a thin (~2-3 nm) oxide layer is formed on the titanium, as is a characteristic surface structure, both of which are judged to be prerequisites for successful osseointegration. However, the surfaces of the components also become covered with a

[0011] layer of impurities, principally residues from cutting liquids which are removed in the subsequent washing stages. During the sterilization in the glass ampoule, the thickness of the oxide layer increases to the final value, which is 4-6 nm.

[0012] Those surfaces of the finished titanium component (the implant) which come into contact with the biological tissue consist, consequently, of a thin titanium oxide layer ( $TiO_2$ ) which is covered with a contamination layer. The latter consists in the main of adsorbed hydrocarbons from the air, etc., and trace quantities of other, inorganic, substances. The exact composition of the contamination layer is a complex function of the whole history of the preparation of the implant. It is desirable that this contamination layer be removed at the same

[0013] time as the characteristic surface structure is retained, since only this surface structure has been found to give clinical results which have been documented as being successful.

[0014] The object of this invention is to control the surface structure of the titanium component with regard to its characteristics and reproducibility to an even higher degree than is the case in the present method. The intention is to prepare the surface so that existing surface contaminants are removed and that the oxide layer which is present is also removed, i.e. so that the "memory" from preceding preparation steps is erased, after which the desired surface is prepared in a well-controlled manner in a controlled environment.

[0015] In accordance with the invention, the surfaces of a plurality of implants are prepared using a gas-discharge plasma (so-called glow discharge).

[0016] The reason for using this type of surface preparation is that, when utilized correctly, it permits a supe-

rior degree of control, reproducibility and flexibility in the manufacturing procedure as compared with a conventional preparation. This assessment is based partly on experiences from other areas of technology in which plasma and other vacuum-based preparation methods are used in the production. In this context, the most conspicuous example is the manufacture of semiconductor components. The high-grade process control which can be achieved results from the fact that vacuum methods per se are very clean and that they can be carried out in closed systems without being affected by uncontrolled environmental atmospheres. Apart from that which has been stated above, the assessment is also based on straightforward fundamental considerations regarding the advantages of a closed vacuum process, and, additionally, on our own experiments (see below). [0015] The principle of using plasma for preparing surfaces can be described simply with the aid of Fig. 1, which diagrammatically illustrates a plasma reactor (vacuum chamber). The gas-discharge plasma 1 is generated by a high voltage (about 1 kV) being applied across a gas between two electrodes under a low pressure 1.33 - 13.330 pascal (~0.01-100 m Torr). The two electrodes can be constituted by the sample and the vacuum chamber itself, as in the figure. The relatively high electrical field results in the few free electrons, which are always present, being accelerated up to energies which are sufficient to ionize the gas molecules with which they collide. This releases further electrons which, in turn, are accelerated and ionize further gas molecules, etc. The ionized molecules will, in turn, be accelerated by the electrical field and collide with gas molecules (which become ionized) and with the surfaces in the system. Both the ionizing events and the ion-surface collisions create new free electrons, thereby bringing about a "cascade effect", resulting in the degree of ionization of the gas being greatly increased. Finally, an equilibrium is established in which a stable current (due to transport of ions and electrons) flows through the gas. This "controlled" gas discharge is a plasma, and is often called a "glow discharge" since it emits visible light as a consequence of the physical processes which are taking place in it. In that which follows, the term plasma is used, which term must not, however, be confused with the type of hot plasmas which are found, for example, in stars, where the degree of ionization and the temperature are much higher.

[0016] The temperature in a gas-discharge plasma is insignificantly higher than the environmental temperature. In addition to neutral gas molecules, free electrons and ionized gas molecules, the plasma also contains free radicals, metastable conditions and other reactive components. These have a much higher tendency to react with, for example, a surface than do unionized and non-excited molecules.

[0017] Depending on a number of different process parameters (DC or AC voltage, electrode configuration and geometrical design, process gas and pressure,

etc.), a large number of different effects can be produced on the sample surface using the plasma. Most of these applications principally utilize the effects which arise when the electrode surfaces are bombarded with high-energy ions in the plasma.

[0018] In this context, some important processes are:

- (i) Sputtering, which entails the high-energy ions knocking off surface atoms from the surface. This effect can be used, inter alia, for mild (dry) cleaning/sterilizing or for finishing/etching surfaces.
- (ii) Surface reactions between ions and surface, entailing some of the bombarding ions reacting chemically with the surface and forming a layer possessing a new chemical composition. The fact that some of the gas molecules are present in ionized or excited conditions makes them, as a rule, more reactive than corresponding neutral molecules. By varying the type of ions (i.e. the process gas), oxide layers, nitride layers or carbide layers can be made, for example.
- (iii) Implantation of ions, i.e. with some of the incoming ions penetrating the surface layer of the sample and becoming embedded in it. Here too, chemical modifications of the surface layer can be produced.
- (iv) "Activation" of the surface. The ion bombardment breaks bonds in the surface, thereby making the latter especially inclined to react with, or bind to, molecules from the environment. This is often termed giving the surface a high degree of surface energy.
- (v) As a rule, structural changes arise at various levels. These can be defects at the atomic level, changes in microstructure, or even modified surface topography and morphology.

[0019] The abovementioned effects occur to different extents and are associated with each other. By varying the process parameters, the processes which are to dominate can to a certain extent be selected. In other words, the plasma technique is a very versatile method for treating surfaces. The method can also advantageously be combined with a variety of subsequent surface-treatment steps in the same chamber in which the plasma treatment takes place.

[0020] In principle, preparing or modifying (cleaning, sterilizing, oxidizing, nitrating, etc.) implant surfaces using gas-discharge plasma is not novel. The method was proposed and tested as early as the 70s by Baier, inter alia. In recent years, the method has excited greatly increased interest in relation to implants, and the use of a gas-discharge plasma for cleaning dental titanium fixtures, for example, has been proposed in US-PS 5,071,351.

[0021] "Plasma cleaning and related treatments" is also described in J. Biomed. Mater. Res.: Applied Bio-materials, Vol 22, No. A2, 145-158 (1988), Bengt Kasemo and Jukka Lausmaa "Biomaterial and implant surfaces:

On the role of cleanliness, contamination, and preparation procedures" on pages 152-153.

[0022] However, in those instances in which plasma preparation has been used in connection with implants, the potential of the method has not been exploited to the full. The reason for this is as follows:

[0023] During plasma preparation, a reactive surface having high so-called surface energy is formed as a rule. This means that the surface has a strong tendency to bind to itself molecules from the surrounding atmospheres to which it is exposed. In most instances in which plasma preparation has been applied to implant surfaces, conventional and/or commercial plasma equipment has been used in which, after completion of the plasma treatment, the plasma-prepared surface is exposed to uncontrolled atmospheres, signifying that the properties of the plasma-prepared surface can be lost. The contaminating molecules which become bound to the surface are often different types of hydrocarbons and other volatile organic molecules. As an example of how rapidly this contamination takes place, it can be mentioned that at a concentration of a contaminant in the air of 1 ppb ( $10^{-9}$ ) a monolayer of contaminating molecules can be bound to the surface in  $\sim 1$  s. In the case of ppm concentrations, the corresponding time scale is  $\sim 1$  ms.

[0024] Apart from the plasma-prepared surface, in the case of previous commercial plasma equipment, having been exposed to uncontrolled atmospheres after the treatment, the plasma equipment has not been adapted, as far as implants are concerned, to production-scale conditions. Thus, the Harrick Scientific chamber which is described in US-PS 5,071,351 is not suitable when a relatively large number of titanium components are to be prepared during production. For example, divergences in the properties can occur if several implants are being treated simultaneously, due to variations in the different geometrical positions of the plasma in the plasma chamber.

[0025] The object of this invention is to develop a method for preparing metallic implant surfaces, preferably made of titanium, using a gas-discharge plasma, where the equipment and the process parameters have been adapted for large-scale production. At the same time, it is required of the plasma process that the surface properties of the finished product, with regard to chemical composition, oxide thickness and structure, should come within the predetermined characteristics. However, the divergence between individual samples and between different sample batches should be less than in the case of current production. In addition, the process should not introduce new surface contaminants.

[0026] The plasma process should in addition be such that the macroscopic appearance of the surface and the microstructure (topography) of the surface within the interval 10-1000 nm are not altered by the process. In addition, in those instances in which the process includes ampoule sealing, all the components in the pack, and the total final result, should satisfy the official require-

ments for sterility which are in force.

[0027] The invention is based, in this context, on a closed system concept in which the plasma preparation and, where appropriate, the remaining preparation steps, and, where appropriate, the packaging and transport to biological environments, as well, are carried out in accordance with a closed procedure without intermediate exposure to uncontrolled environmental atmospheres. This approach has the very great advantages, as compared with existing processes, that a very high grade of controlled surface structure and reproducibility can be achieved and that the properties of the plasma-prepared surface can be preserved right up to the moment of use. Such controlled removal of the oxide layers is not at all disclosed in US-A-5 071 351.

[0028] In accordance with the invention, the implants are conveyed, after customary machining and any washing procedures which may be necessary, to a vacuum chamber in which the plasma preparation is carried out in two steps, firstly treatment with an inert gas plasma so that existing surface contamination layers as well as oxide layers are removed from the implant surface and then reoxidation, using pure O<sub>2</sub> or O<sub>2</sub> plasma the plasma preparation and any remaining preparation steps which may be necessary, and handling of the implants, being carried out in accordance with a closed procedure without intermediate exposure to uncontrolled environmental atmospheres, and as characterized in claim 1.

[0029] The high-vacuum chamber (the preparation chamber) must have a vacuum performance which corresponds to the stipulated requirements for purity and control in the process. Preferably, the basal pressure should be below  $101.3 \cdot 10^{-6}$  pascal ( $10^{-6}$  mbar.) The plasma process pressure is in the mbar range, except in the case of the thermal oxidation in O<sub>2</sub>, when the pressure can be higher, in the (10-1000 mbar)  $1.013 \cdot 10^3 - 101.3 \cdot 10^3$  pascal range.

[0030] In a first embodiment, the plasma treatment (cleaning and oxidation) complements or replaces a part of the present cleaning, i.e. as a last step before sealing and sterilizing in glass ampoules is carried out.

[0031] Alternatively, the plasma treatment can be coupled together with sterilizing and sealing in glass ampoules. This involves all the steps from final cleaning to sterilization and sealing in glass ampoules being carried out in a closed vacuum system without intermediate exposure to environmental or other uncontrolled atmospheres.

[0032] In that which follows, the invention will be described in more detail in association with the enclosed drawings, which show some examples of how the invention can be applied.

[0033] Figure 1 diagrammatically illustrates a conventional vacuum chamber for plasma treatment,

[0034] Figure 2 diagrammatically shows two alternatives for carrying out plasma treatment of titanium implants,

[0035] Figure 3 consists of a sketch showing the principle of equipment for closed plasma treatment under production-scale conditions, where alt. A shows the case in which the plasma-treatment equipment is coupled to a closed space possessing a controlled atmosphere and in which sterile packaging takes place (for example a so-called glove box, or a sterile bench) and alt. B shows the case in which sterilization and packaging have been incorporated into the plasma equipment,

[0036] Figure 4 is a diagrammatic sketch of the plasma cell itself,

[0037] Figure 5 shows a detail of a cathode and sample holder, and

[0038] Figure 6 shows the principle involved in scaling up the preparation of several samples (titanium components) by multiplying the number of plasma cells.

[0039] The principle of plasma preparation of surfaces has already been described in the introduction in conjunction with Figure 1. The gas-discharge plasma 1 is generated by a high voltage being applied across the gas under a low pressure. The two electrodes are constituted by the sample 2 (the titanium component) and the wall 3 of the vacuum chamber. The vacuum chamber is provided with a gas inlet 4 and a gas outlet to a pump 5.

[0040] Figure 2 shows how the plasma treatment of titanium components can be applied under production scale conditions.

[0041] In accordance with alt. A, plasma treatment is introduced after machining and washing procedures (cleaning and oxidation) as a complement to, or replacement for, the cleaning which was previously effected, i.e. as a last step before mounting, sealing and sterilizing in glass ampoules.

[0042] In accordance with alt. B, sterilization, mounting and sealing in glass ampoules are incorporated into the system for plasma treatment. This involves final cleaning, sterilization and sealing in glass ampoules being carried out in a closed (vacuum) system without any intermediate exposure to uncontrolled environmental atmospheres.

[0043] Between these two alternatives, there are a number of "intermediate levels", depending on how the sample transport between plasma treatment and sterile packaging is effected. For example, alt. A above could be coupled together with glass ampoule sealing in a controlled atmosphere (in a so-called glove box filled with synthetic air or other suitable gas).

[0044] In the subsequent description, it is assumed that a closed procedure is being used, i.e. alt. A above combined with sterile packaging in a controlled atmosphere, or alt. B above.

[0045] The plasma treatment consists principally of two steps. In the first step, the outermost  $\leq 10$  nm of the sample surface are removed using an inert gas plasma. The surface is then reoxidized, using pure  $O_2$  or alternatively  $O_2$  plasma for 0.5-20 minutes, in such a way that the desirable oxide layer is obtained. (In order to

produce further surface modifications, other process gases can be used, such as, for example,  $N_2$  (nitridation),  $H_2O$ , (hydroxylation),  $H_2O_2$ ,  $SO_4/PO_4$ , ions (doping) and monomers (polymer coatings)). Suitable process parameters for cleaning and oxidation can, for example, be:

[0046] Cleaning: pure (>99.999%) argon gas; 0.5-3 kV negative direct current voltage on the sample; 10.13 - 50,65 pascal ( $\sim 0.1$ - $0.5$  mbar) Ar pressure; 1.013-10,13 pascal (0.01-0.1 mbar) s $^{-1}$  Ar flow; resulting in plasma currents of  $\sim 0.5$ - $2$  mA cm $^{-2}$  sample surface; for 0.5-20 minutes.

[0047] Oxidation: thermal oxidation in 101.3 - 101.3·10 $^3$  pascal (1-1000 mbar) pure (>99.99%)  $O_2$  at room temperature for 10 min., alternatively  $O_2$  plasma (0.2-3 kV negative direct current voltage on the sample; 1.013 - 50,65 pascal (0.01-0.5 mbar)  $O_2$  pressure; 1.013-10,13 pascal (0.01-0.1 mbar) s $^{-1}$   $O_2$  flow; resulting in plasma currents of  $\sim 0.5$ - $50$  mA cm $^{-2}$  sample surface; for 0.5-20 minutes).

[0048] The preparation can also advantageously be effected in a combined process by gradually introducing oxidizing gas into the process gas (for example mixing  $O_2$  into Ar) during the final phase of the cleaning.

[0049] Since the plasma treatment is carried out in vacuo 1.013-10,13 pascal (pressure 10 $^{-1}$ -10 $^{-3}$  mbar), the equipment for plasma treatment consists in principle of a vacuum system with components which are necessary for the plasma process and which are vacuum-compatible. From the point of view of cleanliness, it is advantageous if the vacuum system consists of separate chambers which are coupled together via valves and between which the samples can be transferred without the vacuum being broken. The principle of equipment for closed plasma preparation in accordance with the two alternatives is shown diagrammatically in Fig. 3.

[0050] That which the two alternatives have in common is:

40 1. A vacuum chamber (K1) whose function is to sluice the sample into the plasma preparation chamber (K2). K1 is accessible from the atmosphere via a valve V1, and is also coupled to K2 via valve V2. If required, K1 can also be coupled to a gas inlet for flushing gas, in order to maintain a higher degree of cleanliness.

45 2. A vacuum chamber (K2), in which the plasma preparation, and any other preparation steps (for example oxidation or heat-sterilization) which may be necessary, takes place. The plasma chamber contains components which are suitable for the process, for example a plasma electrode, electrical connections, process gas inlet, pressure or flow regulating systems, etc.. The vacuum requirements for this chamber must satisfy the requirements which are stipulated by the plasma process (described below). Due to the fact that a sluicing system is used, this chamber is never exposed to air,

except during maintenance work, and can as a result be maintained at a high degree of cleanliness. 3. A vacuum chamber (K3) which functions as a sluice for discharging the plasma-prepared samples through the valve V4. K3 is coupled to the preparation chamber via valve V3. When the system is assembled linearly, the process takes place continuously, i.e. new samples are conveyed into K1 while a set of samples is plasma-prepared in K2, etc.. If a continuous process is not required, V3, K3 and V4 can be dispensed with. K1 then functions as a sample sluice both for conveying in and conveying out the samples. K3 and V3 can also be dispensed with if V4 is coupled to K1, although in a different direction as compared to V1.

[0051] The implants are expediently conveyed into the system mounted on a cassette or the like 6 (described below), thereby allowing several samples to be treated on each occasion. The cassette is then conveyed between the different chambers through the valves using an appropriate transport system. Depending on which of the alternatives A and B is under consideration, the following system components are also additionally required:

(A)4. In order that a closed process can be effected, the discharge sluice (K3, V4) is coupled directly to a closed space in which a controlled (and, where appropriate, sterile) atmosphere (vacuum, atmospheric pressure or excess pressure) can be maintained. In this volume, sterile packaging and, where appropriate, sterilization, are effected either manually or automatically. This space can be, for example, a so-called glove box which satisfies necessary requirements for sterility. In addition to sterile packaging, a multiplicity of components and functions can advantageously be incorporated into this space, for example UV irradiation for cleaning/sterilizing, admission of a suitable gas atmosphere, and analytical equipment, for example a mass spectrometer, for monitoring the gas composition in the space (and thus also the sterile packaging). It should also be possible to carry out any necessary further preparations of the implant surface in this space under atmospheric pressure or in a liquid. (B)4. Where the sterile packaging step is carried out in the vacuum system, a chamber (K3) and a valve (V3), which is expediently situated between the plasma-preparation chamber and the discharge sluice, are additionally required. This chamber contains the necessary components for carrying out the sterile packaging (manipulators, supply of components for the packaging, equipment for sealing the packaging material, etc.).

[0052] Each sample (titanium component) 2 is plasma-prepared individually in a local plasma cell (Figs. 4

and 5) in which the sample constitutes the cathode (by being coupled to negative high voltage or to an alternating (high) current voltage) 7. A hollow cylinder 8 around the sample functions as the anode (normally earthed).

- 5 The plasma cell can also advantageously function as the local gas inlet 9 around the sample (see Fig. 4). A construction of this type means that the flow of process gas around the sample can be effectively controlled, that the influence of any impurities in the residual gas in the preparation chamber can be minimized, and that a certain cooling effect on the sample can be brought about. The geometry of the cell ensures that the gas-discharge plasma is delimited around the sample. (The geometry of the anode can be adapted in a suitable manner for non-cylindrical samples). This local arrangement allows simple scaling-up using a number of identical plasma cells, with gas flows and plasma conditions otherwise being identical.
- 10 [0053] The sample 2 is mounted on a cathode rod 10, which is electrically shielded by an earthed shield 11 which eliminates discharges outside the cell. In order to avoid short circuits between the earthed shield and the cathode, insulating material 12 is applied at appropriate sites (Fig. 5). In order to minimize the risk of contaminants from the cell, all the components which the sample surface "sees" should be manufactured in the same material as the sample. The titanium grating 13 on the gas inlet has the function of delimiting the plasma and of eliminating contaminants from the material used for manufacturing the gas inlet.
- 15 [0054] Besides this, Figure 4 shows two vacuum flanges 14 and 15 with gas inlet and electrically insulating vacuum lead-through 16 for the high voltage, respectively. A pressure or flow regulator 17 regulates the gas supply.
- 20 [0055] Figure 5 shows the cathode 10 and the sample holder in more detail. The cathode is cylindrical and has a narrow projecting threaded part 18 onto which the titanium component, for example a fixture, is screwed.
- 25 [0056] Figure 6 shows how a multiplicity of titanium components (samples) 2 can be plasma-treated simultaneously by the number of plasma cells being multiplied. Under these circumstances, a matrix of identical cells is assembled whose gas supply is effected by a "branched pipe" 23. The conditions in each individual cell are then identical with that described above for one cell. A large number of samples can thereby be treated simultaneously under identical conditions, permitting high production capacity.

**Claims**

1. A method for preparing the surfaces of a plurality of implants made of a metallic material, preferably titanium, using a gas-discharge plasma with the aim of obtaining a well-defined and reproducible implant surface and wherein the implants (2), after machining and any washing procedures which may be necessary, are conveyed to a vacuum chamber (K2) in which the plasma preparation is carried out with an inert gas plasma so that existing surface contamination layers are removed from the implant surfaces, the plasma preparation and any remaining preparation steps which may be necessary, and handling of the implants, being carried out in accordance with a closed procedure without intermediate exposure to uncontrolled environmental atmospheres, **characterized in that** each implant is plasma prepared individually in a local plasma cell having a geometry that ensures that the gas-discharge plasma is delimited around the implant and that the plasma preparation is carried out in two steps, firstly treatment with said inert gas plasma with 0.5-3 kV negative direct current voltage on the implant for 0.5-20 minutes so that the existing surface contamination layers as well as oxide layers are removed from the implant surfaces and secondly reoxidation, using pure O<sub>2</sub> or alternatively, O<sub>2</sub> plasma for 0.5-20 minutes so that the desirable oxide layer is obtained.
  
2. A method according to claim 1, **characterized in that** the plasma preparation is carried out as a complement to, or replacement for, a part of previously used cleaning procedures, i.e. as a last step before the implants are mounted, sealed and sterilized in glass ampoules.
  
3. A method according to claim 1, **characterized in that** sterilization, mounting and sealing of implants in glass ampoules is incorporated into the plasma preparation system, signifying that final cleaning, final preparation, sterilization and sealing in glass ampoules are carried out in a closed vacuum system without intermediate exposure to uncontrolled atmospheres.
  
4. A method according to claim 1, **characterized in that** a plurality of implants are mounted on a cassette (6), which is conveyed into the vacuum chamber (K2) so that a plurality of implants are prepared simultaneously.
  
5. A method according to claim 4, **characterized in that** the cassette (6) is conveyed into and out of the vacuum chamber (K2) for plasma preparation (the preparation chamber) via one or more sample sluices in the form of vacuum chambers (K1,K3) which

are coupled to the preparation chamber via valves (V2,V3).

**5 Patentansprüche**

1. Verfahren zum Präparieren der Oberflächen einer Mehrzahl von Implantaten aus Metall, vorzugsweise Titan, mittels eines Gasentladungs-Plasmas, um eine wohldefinierte und reproduzierbare Implantatoberfläche zu erhalten, wobei die Implantate (2), nach der maschinellen Bearbeitung und ggf. notwendigen Waschvorgängen, zu einer Vakuumkammer (K2) befördert werden, in welcher die Plasmabehandlung mit einem inerten Gasplasma derart durchgeführt wird, daß vorhandene oberflächliche Kontaminations-Schichten von den Implantatoberflächen entfernt werden, wobei die Plasmabehandlung und etwaige weitere notwendige Behandlungsschritte sowie die Handhabung der Implantate gemäß einem geschlossenen Verfahrensablauf durchgeführt werden, ohne zwischenzeitlich unkontrollierten Umgebungsatmosphären ausgesetzt zu werden, dadurch gekennzeichnet, daß jedes Implantat individuell plasmabehandelt wird in einer örtlichen Plasmazelle mit einer Geometrie, die sicherstellt, daß das Gasentladungsplasma um das Implantat herum begrenzt ist, und daß die Plasmabehandlung in zwei Schritten durchgeführt wird, nämlich zuerst eine Behandlung mit dem inerten Gasplasma bei 0,5 - 3 KV negativer Gleichspannung an dem Implantat während 0,5 - 20 Minuten derart, daß die vorhandenen oberflächlichen Kontaminationsschichten sowie Oxidschichten von den Implantatoberflächen entfernt werden, und zweitens eine Re-Oxidierung mit reinem O<sub>2</sub> oder wahlweise O<sub>2</sub>-Plasma während 0,5 - 20 Minuten derart, daß die gewünschte Oxidschicht erhalten wird.
  
2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Plasmabehandlung durchgeführt wird als eine Ergänzung zu oder ein Ersatz für einen Teil der früher verwendeten Reinigungsvorgänge, d.h. als ein letzter Schritt, bevor die Implantate in Glasampullen montiert, eingesiegelt und sterilisiert werden.
  
3. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die Sterilisierung, Montage und Einschließung von Implantaten in Glasampullen in das Plasmabehandlungssystem integriert ist, d.h. daß die Endreinigung, Endbehandlung, Sterilisierung und Einschließung in Glasampullen in einem geschlossenen Vakuumsystem ohne zwischenzeitliche Exponierung zur unkontrollierten Atmosphäre durchgeführt werden.
  
4. Verfahren nach Anspruch 1,

- dadurch gekennzeichnet, daß eine Mehrzahl von Implantaten auf einer Kassette (6) montiert werden, die in eine Vakuumkammer (K2) gebracht wird derart, daß eine Mehrzahl von Implantaten gleichzeitig präpariert werden.
5. Verfahren nach Anspruch 4,  
dadurch gekennzeichnet, daß die Kassette (6) in die Vakuumkammer (K2) für die Plasmabehandlung (die Behandlungsklammer) und aus ihr heraus bewegt wird über eine oder mehrere Probenschleusen in der Form von Vakuumkammern (K1, K3), die über Ventile (V2, V3) mit der Behandlungskammer gekoppelt sind.
- Revendications**
1. Procédé pour préparer les surfaces d'une pluralité d'implants constitués d'un matériau métallique, de préférence du titane, en utilisant un plasma de décharge gazeuse dans le but d'obtenir une surface d'implant bien définie et pouvant être reproduite et dans lequel les implants (2), après usinage et tout procédé de lavage qui peut être nécessaire, sont transportés dans une chambre sous vide (K2) dans laquelle la préparation de plasma est effectuée à l'aide d'un plasma de gaz inerte de sorte que des couches contaminantes de surface existantes sont éliminées des surfaces d'implant, la préparation de plasma et toute étape de préparation restante qui peut être nécessaire, et la manipulation des implants, qui sont effectuées conformément à un procédé fermé sans exposition intermédiaire à des atmosphères environnementales non-commandées, caractérisé en ce que chaque implant est préparé au plasma de manière individuelle dans une cellule à plasma locale ayant une géométrie qui garantit que le plasma de décharge gazeuse est délimité autour de l'implant et que la préparation de plasma est effectuée en deux étapes, tout d'abord un traitement à l'aide dudit plasma de gaz inerte avec une tension en courant continu négatif de 0,5 à 3 kV sur l'implant pendant 0,5 à 20 minutes de sorte que les couches contaminantes de surface existantes ainsi que les couches d'oxyde sont éliminées des surfaces d'implant et deuxièmement une ré-oxydation, en utilisant O<sub>2</sub> pur ou en variante un plasma de O<sub>2</sub> pendant 0,5 à 20 minutes de sorte que la couche d'oxyde souhaitable soit obtenue.
  2. Procédé selon la revendication 1, caractérisé en ce que la préparation de plasma est effectuée en tant que complément d'une partie de procédés de nettoyage utilisés précédemment, ou en remplacement de ceux-ci, c'est-à-dire en tant que dernière étape avant que les implants soient montés, étanchéifiés et stérilisés dans des ampoules de verre.
  3. Procédé selon la revendication 1, caractérisé en ce que la stérilisation, le montage et l'étanchéification des implants dans des ampoules de verre sont incorporés dans le système de préparation de plasma, signifiant qu'un nettoyage final, une préparation finale, une stérilisation et une étanchéification dans des ampoules de verre sont effectués dans un système sous vide fermé sans exposition intermédiaire à des atmosphères non-commandées.
  4. Procédé selon la revendication 1, caractérisé en ce qu'une pluralité d'implants sont montés sur une cassette (6), qui est transportée dans la chambre sous vide (K2) de sorte qu'une pluralité d'implants sont préparés simultanément.
  5. Procédé selon la revendication 4, caractérisé en ce que la cassette (6) est transportée à l'intérieur et à l'extérieur de la chambre sous vide (K2) pour une préparation de plasma (la chambre de préparation) via une ou plusieurs canalisations d'échantillon ayant la forme de chambres sous vide (K1, K3) qui sont couplées à la chambre de préparation via des vannes (V2, V3).

Fig. 1

PRIOR ART

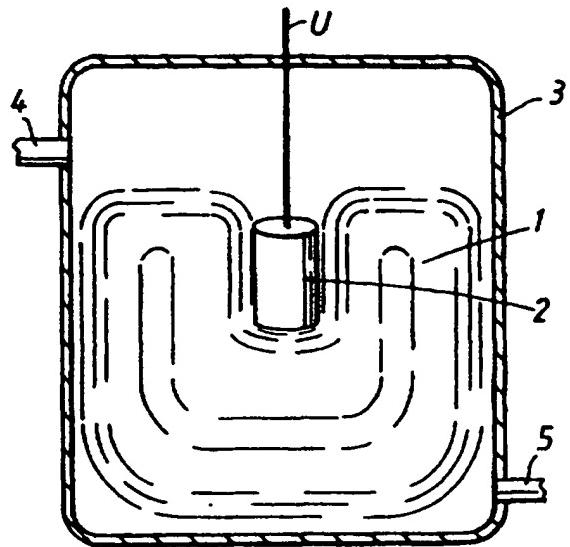
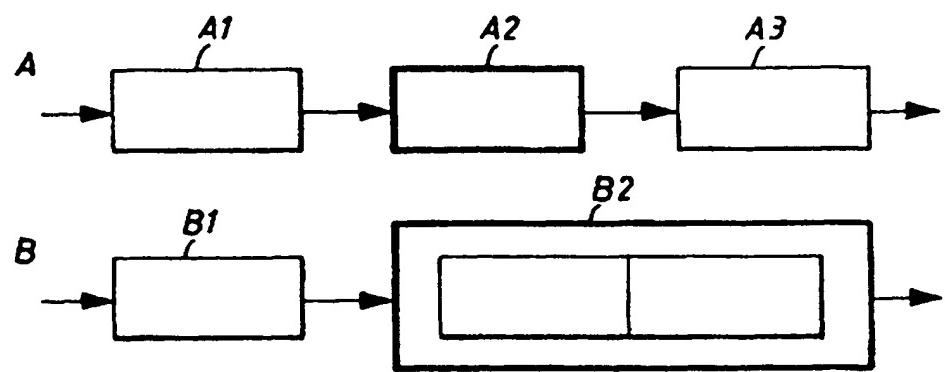


Fig. 2



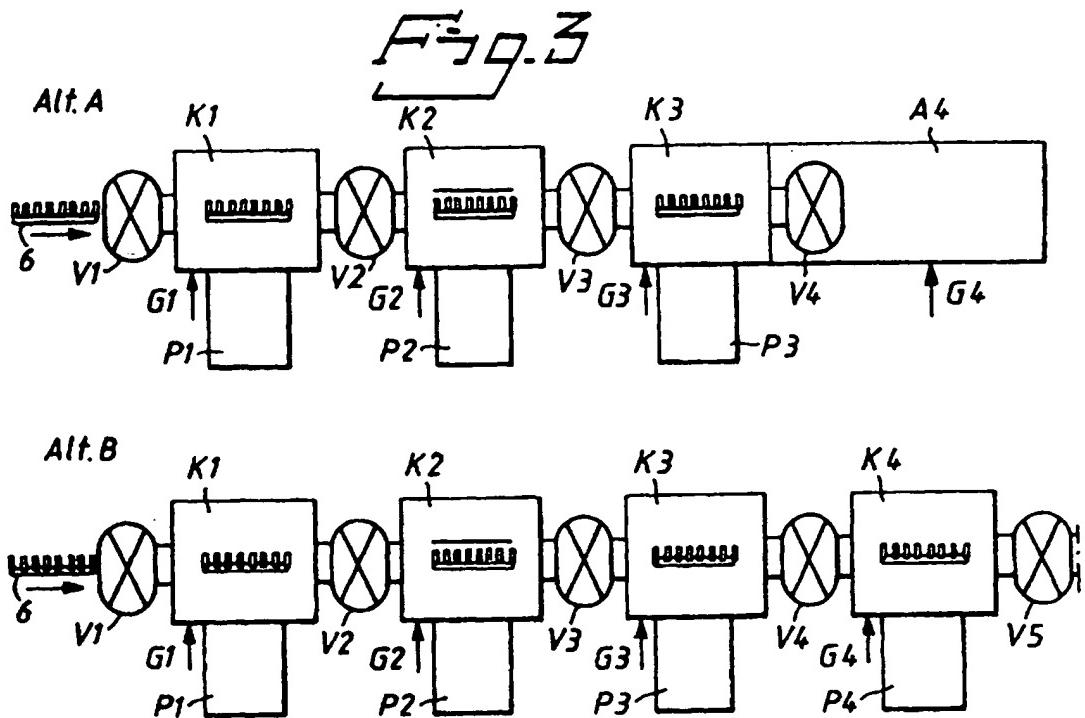
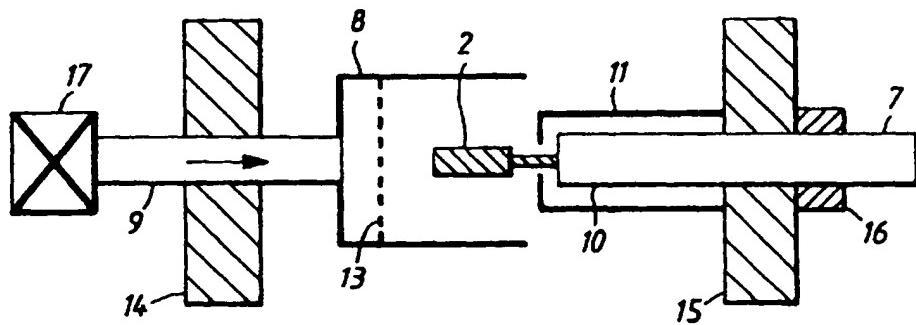
*F-70.4*

Fig. 5

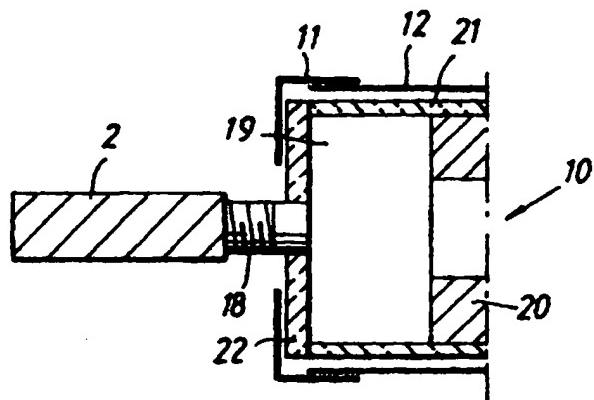


Fig. 6

